

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 06-126851  
(43)Date of publication of application : 10.05.1994

---

(51)Int.Cl. B29C 67/22  
B29C 39/38  
B29C 39/42  
C08J 9/06  
// B29K 23:00  
B29K105:04  
B29K105:24  
C08L 23:00

---

(21)Application number : 04-300429 (71)Applicant : INOAC CORP  
(22)Date of filing : 13.10.1992 (72)Inventor : MATSUBARA TATSUO

---

**(54) PRODUCTION OF POLYOLEFIN FOAM**

**(57)Abstract:**

**PURPOSE:** To provide a method for forming the polyolefin foam rich in compressive stress and small in permanent compressive strain.

**CONSTITUTION:** The method for producing polyolefin foam comprises filling a primary metal die with a foaming composition containing polyolefin, a cross-linking agent and a foaming agent, heating the primary metal die under a pressure of at least 50kg/cm<sup>2</sup> to decompose a portion of the foaming agent at such a decomposition ratio as to satisfy the formula expressed as regards a first foaming process, i.e. foaming agent decomposition %=(9 to 12) × (100/final expansion ratio), to thereby induce the foaming, thereafter releasing pressure from the primary metal die maintained at a high temperature to effect a primary expansion of the foaming agent, removing the expanded mass from the primary metal die as an intermediate foam, subsequently placing the intermediate foam into a secondary metal die having the size and shape corresponding to those of a final foam, heating the intermediate foam under normal pressure to decompose the residual foaming and cross-linking agents and effect a secondary expansion thereof, thereby producing the final foam (having an average foam diameter of 250-450μm and a continuous foam ratio of 5-20%).

---

**LEGAL STATUS**

[Date of request for examination] 26.09.1996

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 2872505

[Date of registration] 08.01.1999

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's

[decision of rejection]

[Date of extinction of right]

08.01.2004

Copyright (C); 1998,2003 Japan Patent Office

**\* NOTICES \***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**CLAIMS**

---

**[Claim(s)]**

[Claim 1] Primary metal mold is filled up with the fizz constituent containing polyolefine, a cross linking agent, and a foaming agent. It is this primary metal mold 50kg/cm<sup>2</sup> It heats under the above pressurization. Some above-mentioned foaming agents The 1st process which is primarily decompressed and expanded at the time of elevated-temperature heat, takes out from the above-mentioned primary metal mold, and manufactures middle foam after making it decompose so that it may become the cracking severity with which are satisfied of a bottom type and carrying out induction of the foaming, By then, the thing for which the middle foam obtained at the 1st process of the above is put in in the secondary mold corresponding to the dimension configuration of the last foam, and the above-mentioned middle foam is heated under ordinary pressure the 2nd process which is expanded secondarily and manufactures the last foam while making the remainder of the above-mentioned foaming agent and the above-mentioned cross linking agent disassemble — since — the manufacture approach of the polyolefine foam characterized by becoming.

Foaming agent cracking severity (%) =(9-12) x of the 1st process (the 100-/last expansion ratio)

[Claim 2] The diameter of average air bubbles of the above-mentioned last foam is the manufacture approach of polyolefine foam according to claim 1 that it is 250-450 micrometers, and the rate of continuous foam is 5 - 20%.

[Claim 3] The manufacture approach of the polyolefine foam of each dimension of the length which constitutes the building envelope of the above-mentioned secondary mold, width, and height according to claim 1 or 2 which is all the dimension which made the above-mentioned middle foam small 1 to 10% to each dimension immediately after foaming of the foam which carried out the last foaming.

---

[Translation done.]

## \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

## DETAILED DESCRIPTION

---

### [Detailed Description of the Invention]

#### [0001]

[Industrial Application] This invention relates to the approach the diameter of air bubbles is comparatively greatly excellent in compressive stress with about 250-450 micrometers, and the rate of continuous foam manufactures efficiently polyolefine foam with comparatively small compression set at 5 ~ 20%, if it says in more detail about the manufacture approach of polyolefine foam.

#### [0002]

[Description of the Prior Art] As the manufacture approach of the block foam of polyolefine How (it is hereafter called one-step foaming.) to expand the consistency of at once a request of this admixture by filling up metal mold with the admixture of polyolefin resin, a cross linking agent, and a foaming agent, disassembling the cross linking agent and a foaming agent completely in the state of pressurization and heating generally, and decompressing after that And fill up primary metal mold, and heat admixture under pressurization and it is primarily expanded as indicated by JP,52-8348,B, JP,2-42649,B, etc. Then, the method of heating the middle foam by ordinary pressure, expanding the 2nd order, and obtaining the last foam of a desired consistency (it is hereafter called two-step foaming.) It is known.

[0003] However, in the above-mentioned one-step foaming, deformation arose in the last foam obtained, and when high foam was obtained and it took out from metal mold since the last foam of a request consistency is expanded at once, the crack arose in the foam, and there was a problem that the rate of commercial production became very low. Therefore, two-step foaming was developed that the product yield fall of the above-mentioned one-step foaming should be prevented. By two-step foaming, the factor which reduces product yields, such as deformation and a crack, is removed by not carrying out foaming expansion of the product of predetermined expansion ratio at once, dividing into two steps and carrying out foaming expansion.

#### [0004]

[Problem(s) to be Solved by the Invention] However, by the above-mentioned two-step foaming method, the countless nucleus air bubbles formed by thermal decomposition of a foaming agent in part are grown up into a 70-90-micrometer detailed cel under "primary high pressure at the time of decompressing expansion. Since it is performed by the cellular formation mechanism of growing up this detailed cel into the 100-150-micrometer diameter of average air bubbles by the secondary [ further ] ordinary pressure foaming expansion at homogeneity" The last foam obtained turns into a uniform detailed closed cell object, and, generally such foam has the fault that permanent set is comparatively large, deficiently [ compressive hardness ].

[0005] This invention aims at it being rich in compressive stress, and offering the manufacture approach of the small foam of a permanent compressive strain by solving the above-mentioned trouble, improving the physical properties of the foam in two-step foaming which was excellent in the above-mentioned commercial production yield, and rationalizing the amount of foaming agent decomposition in primary foaming in two-step foaming.

#### [0006]

[Means for Solving the Problem] The manufacture approach of the polyolefine foam of this

invention Primary metal mold is filled up with the fizz constituent containing polyolefine, a cross linking agent, and a foaming agent. It is this primary metal mold 50kg/cm<sup>2</sup> It heats under the above pressurization. Some above-mentioned foaming agents The 1st process which is primarily decompressed and expanded at the time of elevated-temperature heat, takes out from the above-mentioned primary metal mold, and manufactures middle foam after making it decompose so that it may become the cracking severity with which are satisfied of a bottom type and carrying out induction of the foaming, By then, the thing for which the middle foam obtained at the 1st process of the above is put in in the secondary mold corresponding to the dimension configuration of the last foam, and the above-mentioned middle foam is heated under ordinary pressure the 2nd process which is expanded secondarily and manufactures the last foam while making the remainder of the above-mentioned foaming agent and the above-mentioned cross linking agent disassemble — since — it is characterized by becoming.

Foaming agent cracking severity (%) =(9-12) x of the 1st process (the 100-/last expansion ratio) [0007] In this invention with "polyolefine" For example, the high pressure process usually marketed, Polyethylene, ethylene propylene rubber which were manufactured by the medium-voltage method or the low voltage method, An ethylene-butene copolymer, an ethylene-vinylacetate copolymer, ethylene, and methyl, Ethyl, propyl, or a copolymer with each acrylic ester (the content of this ester; less than [ 45 mol % ]) of butyl, Or mixture with these things chlorinated to 60 % of the weight of chlorine content, respectively and also these two or more sorts of mixture, these and isotactic polypropylene, or atactic polypropylene etc. is said.

[0008] It has the decomposition temperature more than the flow beginning temperature of polyolefine at least in the above-mentioned polyolefine, and is decomposed by heating, and the "cross linking agent" said to this invention generates an uncombined radical, and says the organic peroxide which is the radical generating agent which produces and cheats out of a crosslinking bond between the molecule. For example, it is dicumyl peroxide, 2, 5-dimethyl -2, 5-screw-tertiary butyl peroxyhexane, 1, and 3-screw-tertiary peroxy-isopropylbenzene etc. The "foaming agent" said to this invention means what has the decomposition temperature more than the flow beginning temperature of the above-mentioned polyolefine, for example, is an AZOJI carvone amide, dinitrosopentamethylenetetramine, etc.

[0009] moreover, metallic oxides, such as a compound which uses a urea as a principal component in this invention in order to control a foaming condition, a zinc oxide, and lead oxide, — low-grade or a higher fatty acid — or foaming assistants, such as low-grade or a metal salt of a higher fatty acid, etc. can be added. Furthermore, carbon black, a zinc white, titanium oxide, and other compounding agents in ordinary use can also be added for a physical-properties improvement.

[0010] In this invention, the pressure in a primary expansion process is "being above 50kg/cm<sup>2</sup>." The pressure of this primary metal mold is 50kg/cm<sup>2</sup>. In the following, when it considers as the foaming agent cracking severity of this invention, since it becomes the conditions which expand foam to the neighborhood 10 times, and the leak of foam arises from primary metal mold at the time of foaming expansion, it becomes the cause of deformation of primary foam and this causes decline in the rate of commercial production further, it is not desirable. Moreover, secondary foaming in the 2nd process is performed under ordinary pressure, and it is usually 20kg/cm<sup>2</sup>. It will be processed under the following pressures. Furthermore, it is because making foaming agent cracking severity of the 1st process into x(9-12) (100 / last expansion ratio) % has a diameter of air bubbles comparatively as large as 250-450 micrometers by doing in this way, it is rich in the foam which has the rate of continuous foam which is 5 - 20%, nothing, and compressive stress and the small product of permanent compressive strain is obtained.

[0011] Furthermore, although there is the approach of adjusting the addition of foaming assistants, such as a metallic oxide, as a means to adjust the amount of decomposition of a foaming agent, as an easiest and positive approach, about whenever [ stoving temperature / of primary foaming ], it is comparatively set as low temperature and there is the about 130-170-degree C approach of controlling by the heating time. Moreover, it is important for whenever [ in secondary foaming / stoving temperature ] to decompose completely and to make a foaming agent foam, and it is desirable to set up in the range which does not have a bad influence on

polyolefine, it is about 160-190 degrees C, and the heating time is usually a 20 - 60-minute about room.

[0012] The above "a secondary mold" has a non-sealing building envelope so that it may not be used under pressurization with closed mold like metal mold. And in case the secondary middle foam expands in this building envelope, it has the structure which can eliminate to the exterior the air which remains in a building envelope by the expansion pressure of foam, and the stoma which makes the suitable mold face of a secondary mold usually open the above-mentioned building envelope and an external ambient atmosphere for free passage is prepared in each 1-2 fields.

[0013] Moreover, the dimension configuration of this secondary type of building envelope is equivalent to the dimension configuration of the last foam. this "corresponding" abbreviation — it is the semantics made into an analog and, thereby, three-dimension expansion of foam can be made to cause in homogeneity as much as possible In connection with this, generating of the irregularity by the contraction spots of the front face of the last foam and a dimension error can be prevented by contraction with the passage of time.

[0014] Moreover, it is desirable that each is [ of each dimension of the length which constitutes the building envelope of the above-mentioned secondary mold, width, and height ] the dimension which made the above-mentioned middle foam small 1 to 10% to each dimension immediately after foaming of the foam which carried out the last foaming. in addition, the case where it is the solid configuration by which the concept of this is not clearly carried out to "the length, the width, and the height" in this case — three dimensions —like — seeing — abbreviation — an analog — it uses for the semantics of a \*\*. While shaping becomes possible as the configuration of this building envelope by the last foam which carried out foaming expansion by making this small 1 to 10% being contacted and forced on homogeneity by that self-expansion force without the place left on the wall surface of this building envelope, the variation in the last foam configuration becomes small. In addition, the heat transfer effectiveness in a heating process (cooling process in the secondary mold performed if needed) also improves.

[0015] In addition, a building envelope dimension becomes insufficient [ less than 1% / the thrust of this foam to this building envelope internal surface by the self-expansion force of foam ], when not small, a foam front face does not become irregularity, or a corner is not formed, and while becoming that it is hard to be fabricated as secondary metal mold, heating effectiveness also gets worse. On the other hand, when it is made small exceeding 10%, even if foam contracts by subsequent cooling, more fairly [ \*\* outside foam ] in addition than this space inside dimension greatly, drawing of the foam from the mold for cooling shaping becomes difficult, or the center section of foam comes floating at the time of disconnection of the mold for cooling, and it becomes the cause of deformation crack generating.

[0016]

[Function] In this invention, since cracking severity of the foaming agent in primary foaming processes is made into the cracking severity with which are satisfied of a front type, the number density of the nucleus air bubbles formed at primary foaming processes at the time of foaming agent decomposition becomes high, and can make it thin, spacing, i.e., the cellular wall, between nucleus air bubbles. Thus, conjointly, the explosive expansion and \*\* which are caused at the time of decompressing can set like the detailed cel growth fault at the time of the explosive expansion, and can carry out generating induction of the destruction of a cellular wall to the cellular wall formed in part. Consequently, it becomes possible for detailed cels to gather and to weld to the cellular wall with which the strand of the cellular wall destroyed while cell size became large remained, to reinforce the cellular wall, and to be rich in compressive stress, and to offer foam with a comparatively small permanent set. For example, the foaming agent cracking severity in the primary process of the case of one 15 times the last expansion ratio of this is 60 - 80%, and at less than 60%, the diameter of average air bubbles becomes small too much, and it becomes deficient in compressive stress, and on the other hand, when it exceeds 80%, deformation of the primary secondary foam and a crack will produce it. That is, in this invention, since foaming cracking severity of the 1st process is made into the suitable predetermined range, the foam which has the rate of continuous foam which the diameter of air bubbles is

comparatively large, and is 5 – 20% can be manufactured with 250–450 micrometers, therefore it can be rich in compressive stress, and the small product of permanent compressive strain can be obtained.

[0017]

[Example] Hereafter, an example explains this invention concretely.

Examples 1–2 and one to example of comparison 3 this example mainly change various heating time of primary foaming, change the foaming agent cracking severity in primary foaming processes, and manufacture the last foam. The constituent which becomes the polyethylene 100 weight section (henceforth the section) of a melt index 1.0 from the AZOJI carvone amide 5 section, the dicumyl peroxide 2 section, the zinc–oxide 0.5 section, and the white light oil 5 section was kneaded on the roll with a skin temperature of 100 degrees C, and admixture was obtained. Primary metal mold (410mmx410mmx40mm) was filled up with 6kg of this admixture, and it was processed under the conditions (heating was performed by making thermal passage flow through steam.) of the primary process shown in Table 1, and subsequently to the time of elevated-temperature heat, it decompressed and the middle foam which carried out foaming expansion was taken out in predetermined magnitude. Then, this was put into the predetermined secondary mold (building–envelope dimension: 1000mmx1000mmx100mm), it was processed under the conditions (heating was performed by making thermal passage flow through steam.) of the secondary process shown in Table 1, and the last foam 15 times the expansion ratio of this was manufactured.

[0018]

[Table 1]

表 1

	実施例1	実施例2	比較例 1	比較例 2	比較例3
1次圧力 (kg/cm <sup>2</sup> )	70	70	70	70	* 40
1次加熱温度 (℃)	160	160	160	160	160
1次加熱時間 (分)	30	35	28	37	35
1次発泡工程での発泡剤分解率	60.0	80.0	* 50.0	* 90.0	80.0
加工数	100	100	100	100	100
一次金型での発泡体割れ	0	0	0	0	* 19
1次発泡体の変形・割れ	0	0	0	* 12	* 14
2次発泡体の変形・割れ	0	0	0	* 9	* 11
平均気泡径 (μm)	270	450	* 120	470	420
25%圧縮応力 (kg/cm <sup>2</sup> )	1.12	1.21	* 0.89	1.23	1.23
連泡率 (%)	7	19	* 2	19	16
25%圧縮永久ひずみ (%)	2.7	2.0	* 4.2	2.3	2.1

[0019] And 100 foam per each processing conditions was made, and it evaluated [ the diameter of average air bubbles of each last foam, 25% compressive stress, the rate of continuous foam, and ] about 25% compression set in deformation of the foam leak and primary foam in the primary metal mold at that time, and the last foam (secondary foam), the crack, and the list, and measured in them, and the result was also written together to Table 1. In addition, it separates from the figure which \* mark attached from this invention range among Table 1. Moreover, the unit of the figure of the column "be transformed and the primary secondary foam should break" is a number among this table. [ the "foam leak in primary metal mold" column and ]

[0020] Measurement of the diameter of average air bubbles measures the diameter of 100 air bubbles per each foam, and shows it by the average. Measurement of 25% compressive stress is JIS. It carried out by the approach of K6767. When the value acquired by \*\*\* measurement is large, compressive stress becomes large and will be rich in compressive stress. Moreover, measurement of the rate of continuous foam (%) was performed based on the Ayr pycnometer method (ASTM D2856). Measurement of 25 more% compression set is JIS. K6767 performed.

[0021] Moreover, the last foam obtained in the example 1 in addition to the physical properties shown in Table 1 995-1000mm (the difference of maximum and the minimum value is 5mm) and thickness were almost the same as the configuration of the building envelope of a secondary

mold, the periphery front face exceeded [ thickness ] in 98–101mm (the difference of maximum and the minimum value is 3mm), and abbreviation homogeneity at smooth nature, and 0.06g /, length cc, and width had the very beautiful consistency.

[0022] Since primary cracking severity is as small as 50% in the example 1 of a comparison, although there are not a leak of foam and deformation according to the result of Table 1, the diameter of average air bubbles is too (it is not a desired thing.) small, 25% compressive stress is small, and the rate of continuous foam is also low, and 25% compression set is also large.

Although it was satisfied with the examples 2 and 3 of a comparison of the physical properties of the last foam, the leak of foam and deformation produced all. On the other hand, in the examples 1 and 2, there is such no fault and the efficiently excellent engine–performance article has been manufactured.

[0023] In examples 3–4 and four to example of comparison 5 this example, the relation between a secondary molded dimension and the foam dimension immediately after the completion of secondary foaming expansion is examined. That is, it is processed on the same conditions as an example 1 using primary metal mold with the predetermined molded dimension which shows the specified quantity (shown in Table 2.) of the same admixture as an example 1 in Table 2.

Furthermore, the same conditions as an example 1 and a facility performed secondary foaming. In addition, the magnitude of a secondary molded dimension is shown in Table 2. These results are also shown in Table 2.

[0024]

[Table 2]

表 2

		実施例3	実施例4	比較例4	比較例5	
1次型発泡性混和物仕込量 (Kg)		5. 5	6. 7	5. 2	7. 1	
一次型寸法 [A] (mm)	縦	398	425	391	434	
	横	398	425	391	434	
	高さ	39	41	38	42	
二次型寸法 [B] (mm)	縦	1000	1000	1000	1000	
	横	1000	1000	1000	1000	
	高さ	100	100	100	100	
2次発泡膨張完了直後の発泡体寸法 [C] (mm)	縦	1018	1087	1002	1123	
	横	1025	1090	993	1111	
	高さ	102	109	100	112	
$\frac{\text{二次型縮小率} (\%)}{[C] - [B]}$	縦	1. 8	8. 0	2. 0	11. 0*	
	横	2. 4	8. 3	-7. 0*	10. 0	
	高さ	2. 0	8. 3	0*	10. 7*	
	平均	2. 1	8. 2	-1. 7*	10. 6*	
最終発泡体寸法 バラツキ (mm)	縦	最大値	970	1038	958	
		最小値	964	1035	949	
		差	6	3	9	
	横	最大値	975	1039	959	
		最小値	972	1037	940	
		差	3	2	19	
	高さ	最大値	98	106	95	
		最小値	95	103	88	
		差	3	3	7	
最終発泡体の取り出しの容易性		容易	容易	容易	困難	
最終発泡体の割れ発生の有無		無し	無し	無し	有り	

[0025] In Table 2, the foam dimension immediately after the completion of secondary foaming expansion [C] is compulsorily taken out from metal mold, without cooling the foam immediately after the completion of secondary foaming, measures the dimension of three places each about each of length, width, and height, and expresses it with the average. Moreover, the variation in the last foam dimension measures the maximum part and the minimum part about the length of the foam after cool down ready, width, and height within a secondary mold, respectively, and expresses them with the difference. It separates from the numeric value which attached \* mark

in Table 2 from the range of the 3rd invention.

[0026] According to the above result, as shown in Table 2, in the examples 4 and 5 of a comparison with unsuitable secondary mold reduction percentage, the dimension variation of the last foam was large. Moreover, in the example 5 of a comparison, the crack with a die length [ of 200mm ] and a depth of 10mm occurred on the foam top face, and drawing from the mold of the last foam is stiff, and needed to take out fairly by force. On the other hand, in the examples 1, 3, and 4, there were no above faults (it is very few although there is variation.), and they were good. In addition, in this invention, it is not restricted to what is shown in the above-mentioned concrete example, but can consider as the example variously changed within the limits of this invention according to the purpose and the application.

[0027]

[Effect of the Invention] It can be rich in the foam which has a rate of continuous foam with it in this invention according to the manufacture approach, i.e., compressive stress, and the last foam with few permanent sets can be obtained easily. [ the large and diameter of air bubbles, and ] [ comparatively moderate ] Moreover, according to the manufacture approach of having the range where secondary mold reduction percentage (difference of the foaming dimension immediately after this completion of secondary foaming expansion and a secondary molded dimension over the foaming dimension immediately after the completion of secondary foaming expansion) is suitable, while also being able to make dimension variation of the last foam very small, it does not need to take out the last foam by force, either and can take it out from a secondary mold easily.

---

[Translation done.]

**\* NOTICES \***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**TECHNICAL FIELD**

---

[Industrial Application] This invention relates to the approach the diameter of air bubbles is comparatively greatly excellent in compressive stress with about 250–450 micrometers, and the rate of continuous foam manufactures efficiently polyolefine foam with comparatively small compression set at 5 – 20%, if it says in more detail about the manufacture approach of polyolefine foam.

---

[Translation done.]

**\* NOTICES \***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**PRIOR ART**

[Description of the Prior Art] As the manufacture approach of the block foam of polyolefine How (it is hereafter called one-step foaming.) to expand the consistency of at once a request of this admixture by filling up metal mold with the admixture of polyolefin resin, a cross linking agent, and a foaming agent, disassembling the cross linking agent and a foaming agent completely in the state of pressurization and heating generally, and decompressing after that And fill up primary metal mold, and heat admixture under pressurization and it is primarily expanded as indicated by JP,52-8348,B, JP,2-42649,B, etc. Then, the method of heating the middle foam by ordinary pressure, expanding the 2nd order, and obtaining the last foam of a desired consistency (it is hereafter called two-step foaming.) It is known.

[0003] However, in the above-mentioned one-step foaming, deformation arose in the last foam obtained, and when high foam was obtained and it took out from metal mold since the last foam of a request consistency is expanded at once, the crack arose in the foam, and there was a problem that the rate of commercial production became very low. Therefore, two-step foaming was developed that the product yield fall of the above-mentioned one-step foaming should be prevented. By two-step foaming, the factor which reduces product yields, such as deformation and a crack, is removed by not carrying out foaming expansion of the product of predetermined expansion ratio at once, dividing into two steps and carrying out foaming expansion.

---

[Translation done.]

**\* NOTICES \***

JPO and NCIPPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**EFFECT OF THE INVENTION**

---

[Effect of the Invention] It can be rich in the foam which has a rate of continuous foam with it in this invention according to the manufacture approach, i.e., compressive stress, and the last foam with few permanent sets can be obtained easily. [ the large and diameter of air bubbles, and ] [ comparatively moderate ] Moreover, according to the manufacture approach of having the range where secondary mold reduction percentage (difference of the foaming dimension immediately after this completion of secondary foaming expansion and a secondary molded dimension over the foaming dimension immediately after the completion of secondary foaming expansion) is suitable, while also being able to make dimension variation of the last foam very small, it does not need to take out the last foam by force, either and can take it out from a secondary mold easily.

---

[Translation done.]

**\* NOTICES \***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**TECHNICAL PROBLEM**

---

[Problem(s) to be Solved by the Invention] However, by the above-mentioned two-step foaming method, the countless nucleus air bubbles formed by thermal decomposition of a foaming agent in part are grown up into a 70-90-micrometer detailed cel under "primary high pressure at the time of decompressing expansion. Since it is performed by the cellular formation mechanism of growing up this detailed cel into the 100-150-micrometer diameter of average air bubbles by the secondary [ further ] ordinary pressure foaming expansion at homogeneity" The last foam obtained turns into a uniform detailed closed cell object, and, generally such foam has the fault that permanent set is comparatively large, deficiently [ compressive hardness ].

[0005] This invention aims at it being rich in compressive stress, and offering the manufacture approach of the small foam of a permanent compressive strain by solving the above-mentioned trouble, improving the physical properties of the foam in two-step foaming which was excellent in the above-mentioned commercial production yield, and rationalizing the amount of foaming agent decomposition in primary foaming in two-step foaming.

---

[Translation done.]

## \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

MEANS

[Means for Solving the Problem] The manufacture approach of the polyolefine foam of this invention Primary metal mold is filled up with the fizz constituent containing polyolefine, a cross linking agent, and a foaming agent. It is this primary metal mold 50kg/cm<sup>2</sup> It heats under the above pressurization. Some above-mentioned foaming agents The 1st process which is primarily decompressed and expanded at the time of elevated-temperature heat, takes out from the above-mentioned primary metal mold, and manufactures middle foam after making it decompose so that it may become the cracking severity with which are satisfied of a bottom type and carrying out induction of the foaming. By then, the thing for which the middle foam obtained at the 1st process of the above is put in in the secondary mold corresponding to the dimension configuration of the last foam, and the above-mentioned middle foam is heated under ordinary pressure the 2nd process which is expanded secondarily and manufactures the last foam while making the remainder of the above-mentioned foaming agent and the above-mentioned cross linking agent disassemble — since — it is characterized by becoming.

Foaming agent cracking severity (%) =(9-12) x of the 1st process (the 100-/last expansion ratio)

[0007] In this invention with "polyolefine" For example, the high pressure process usually marketed, Polyethylene, ethylene propylene rubber which were manufactured by the medium-voltage method or the low voltage method, An ethylene-butene copolymer, an ethylene-vinylacetate copolymer, ethylene, and methyl, Ethyl, propyl, or a copolymer with each acrylic ester (the content of this ester; less than [ 45 mol % ]) of butyl, Or mixture with these things chlorinated to 60 % of the weight of chlorine content, respectively and also these two or more sorts of mixture, these and isotactic polypropylene, or atactic polypropylene etc. is said.

[0008] It has the decomposition temperature more than the flow beginning temperature of polyolefine at least in the above-mentioned polyolefine, and is decomposed by heating, and the "cross linking agent" said to this invention generates an uncombined radical, and says the organic peroxide which is the radical generating agent which produces and cheats out of a crosslinking bond between the molecule. For example, it is dicumyl peroxide, 2, 5-dimethyl-2, 5-screw-tertiary butyl peroxyhexane, 1, and 3-screw-tertiary peroxy-isopropylbenzene etc. The "foaming agent" said to this invention means what has the decomposition temperature more than the flow beginning temperature of the above-mentioned polyolefine, for example, is an AZOJI carvone amide, dinitrosopentamethylenetetramine, etc.

[0009] moreover, metallic oxides, such as a compound which uses a urea as a principal component in this invention in order to control a foaming condition, a zinc oxide, and lead oxide, — low-grade or a higher fatty acid — or foaming assistants, such as low-grade or a metal salt of a higher fatty acid, etc. can be added. Furthermore, carbon black, a zinc white, titanium oxide, and other compounding agents in ordinary use can also be added for a physical-properties improvement.

[0010] In this invention, the pressure in a primary expansion process is "being above 50kg/cm<sup>2</sup>." The pressure of this primary metal mold is 50kg/cm<sup>2</sup>. In the following, when it considers as the foaming agent cracking severity of this invention, since it becomes the conditions which expand foam to the neighborhood 10 times, and the leak of foam arises from primary metal mold at the time of foaming expansion, it becomes the cause of deformation of primary foam and this causes

decline in the rate of commercial production further, it is not desirable. Moreover, secondary foaming in the 2nd process is performed under ordinary pressure, and it is usually 20kg/cm<sup>2</sup>. It will be processed under the following pressures. Furthermore, it is because making foaming agent cracking severity of the 1st process into x(9-12) (100 / last expansion ratio) % has a diameter of air bubbles comparatively as large as 250-450 micrometers by doing in this way, it is rich in the foam which has the rate of continuous foam which is 5 - 20%, nothing, and compressive stress and the small product of permanent compressive strain is obtained.

[0011] Furthermore, although there is the approach of adjusting the addition of foaming assistants, such as a metallic oxide, as a means to adjust the amount of decomposition of a foaming agent, as an easiest and positive approach, about whenever [ stoving temperature / of primary foaming ], it is comparatively set as low temperature and there is the about 130-170-degree C approach of controlling by the heating time. Moreover, it is important for whenever [ in secondary foaming / stoving temperature ] to decompose completely and to make a foaming agent foam, and it is desirable to set up in the range which does not have a bad influence on polyolefine, it is about 160-190 degrees C, and the heating time is usually a 20 - 60-minute about room.

[0012] The above "a secondary mold" has a non-sealing building envelope so that it may not be used under pressurization with closed mold like metal mold. And in case the secondary middle foam expands in this building envelope, it has the structure which can eliminate to the exterior the air which remains in a building envelope by the expansion pressure of foam, and the stoma which makes the suitable mold face of a secondary mold usually open the above-mentioned building envelope and an external ambient atmosphere for free passage is prepared in each 1-2 fields.

[0013] Moreover, the dimension configuration of this secondary type of building envelope is equivalent to the dimension configuration of the last foam. this "corresponding" abbreviation — it is the semantics made into an analog and, thereby, three-dimension expansion of foam can be made to cause in homogeneity as much as possible In connection with this, generating of the irregularity by the contraction spots of the front face of the last foam and a dimension error can be prevented by contraction with the passage of time.

[0014] Moreover, it is desirable that each is [ of each dimension of the length which constitutes the building envelope of the above-mentioned secondary mold, width, and height ] the dimension which made the above-mentioned middle foam small 1 to 10% to each dimension immediately after foaming of the foam which carried out the last foaming. in addition, the case where it is the solid configuration by which the concept of this is not clearly carried out to "the length, the width, and the height" in this case — three dimensions —like — seeing — abbreviation — an analog — it uses for the semantics of a \*\*. While shaping becomes possible as the configuration of this building envelope by the last foam which carried out foaming expansion by making this small 1 to 10% being contacted and forced on homogeneity by that self-expansion force without the place left on the wall surface of this building envelope, the variation in the last foam configuration becomes small. In addition, the heat transfer effectiveness in a heating process (cooling process in the secondary mold performed if needed) also improves.

[0015] In addition, a building envelope dimension becomes insufficient [ less than 1% / the thrust of this foam to this building envelope internal surface by the self-expansion force of foam ], when not small, a foam front face does not become irregularity, or a corner is not formed, and while becoming that it is hard to be fabricated as secondary metal mold, heating effectiveness also gets worse. On the other hand, when it is made small exceeding 10%, even if foam contracts by subsequent cooling, more fairly [ \*\* outside foam ] in addition than this space inside dimension greatly, drawing of the foam from the mold for cooling shaping becomes difficult, or the center section of foam comes floating at the time of disconnection of the mold for cooling, and it becomes the cause of deformation crack generating.

---

[Translation done.]

**\* NOTICES \***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**OPERATION**

---

**[Function]** In this invention, since cracking severity of the foaming agent in primary foaming processes is made into the cracking severity with which are satisfied of a front type, the number density of the nucleus air bubbles formed at primary foaming processes at the time of foaming agent decomposition becomes high, and can make it thin, spacing, i.e., the cellular wall, between nucleus air bubbles. Thus, conjointly, the explosive expansion and \*\* which are caused at the time of decompressing can set like the detailed cel growth fault at the time of the explosive expansion, and can carry out generating induction of the destruction of a cellular wall to the cellular wall formed in part. Consequently, it becomes possible for detailed cels to gather and to weld to the cellular wall with which the strand of the cellular wall destroyed while cell size became large remained, to reinforce the cellular wall, and to be rich in compressive stress, and to offer foam with a comparatively small permanent set. For example, the foaming agent cracking severity in the primary process of the case of one 15 times the last expansion ratio of this is 60 – 80%, and at less than 60%, the diameter of average air bubbles becomes small too much, and it becomes deficient in compressive stress, and on the other hand, when it exceeds 80%, deformation of the primary secondary foam and a crack will produce it. That is, in this invention, since foaming cracking severity of the 1st process is made into the suitable predetermined range, the foam which has the rate of continuous foam which the diameter of air bubbles is comparatively large, and is 5 – 20% can be manufactured with 250–450 micrometers, therefore it can be rich in compressive stress, and the small product of permanent compressive strain can be obtained.

[0017]

---

[Translation done.]

**\* NOTICES \***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**EXAMPLE**

---

[Example] Hereafter, an example explains this invention concretely. Examples 1-2 and one to example of comparison 3 this example mainly change various heating time of primary foaming, change the foaming agent cracking severity in primary foaming processes, and manufacture the last foam. The constituent which becomes the polyethylene 100 weight section (henceforth the section) of a melt index 1.0 from the AZOJI carvone amide 5 section, the dicumyl peroxide 2 section, the zinc-oxide 0.5 section, and the white light oil 5 section was kneaded on the roll with a skin temperature of 100 degrees C, and admixture was obtained. Primary metal mold (410mmx410mmx40mm) was filled up with 6kg of this admixture, and it was processed under the conditions (heating was performed by making thermal passage flow through steam.) of the primary process shown in Table 1, and subsequently to the time of elevated-temperature heat, it decompressed and the middle foam which carried out foaming expansion was taken out in predetermined magnitude. Then, this was put into the predetermined secondary mold (building-envelope dimension: 1000mmx1000mmx100mm), it was processed under the conditions (heating was performed by making thermal passage flow through steam.) of the secondary process shown in Table 1, and the last foam 15 times the expansion ratio of this was manufactured.

[0018]

[Table 1]

表 1

	実施例1	実施例2	比較例 1	比較例 2	比較例3
1次圧力 (kg/cm <sup>2</sup> )	70	70	70	70	* 40
1次加熱温度 (℃)	160	160	160	160	160
1次加熱時間 (分)	30	35	28	37	35
1次発泡工程での発泡剤分解率	60. 0	80. 0	* 50. 0	* 90. 0	80. 0
加工N数	100	100	100	100	100
一次金型での発泡体洩れ	0	0	0	0	* 19
1次発泡体の変形・割れ	0	0	0	* 12	* 14
2次発泡体の変形・割れ	0	0	0	* 9	* 11
平均気泡径 (μm)	270	450	* 120	470	420
25%圧縮応力 (kg/cm <sup>2</sup> )	1. 12	1. 21	* 0. 89	1. 23	1. 23
連泡率 (%)	7	19	* 2	19	16
25%圧縮永久ひずみ (%)	2. 7	2. 0	* 4. 2	2. 3	2. 1

[0019] And 100 foam per each processing conditions was made, and it evaluated [ the diameter of average air bubbles of each last foam, 25% compressive stress, the rate of continuous foam, and ] about 25% compression set in deformation of the foam leak and primary foam in the primary metal mold at that time, and the last foam (secondary foam), the crack, and the list, and measured in them, and the result was also written together to Table 1. In addition, it separates from the figure which \* mark attached from this invention range among Table 1. Moreover, the unit of the figure of the column "be transformed and the primary secondary foam should break" is a number among this table. [ the "foam leak in primary metal mold" column and ]

[0020] Measurement of the diameter of average air bubbles measures the diameter of 100 air bubbles per each foam, and shows it by the average. Measurement of 25% compressive stress is JIS. It carried out by the approach of K6767. When the value acquired by \*\*\* measurement is large, compressive stress becomes large and will be rich in compressive stress. Moreover, measurement of the rate of continuous foam (%) was performed based on the Ayr pycnometer method (ASTM D2856). Measurement of 25 more% compression set is JIS. K6767 performed.

[0021] Moreover, the last foam obtained in the example 1 in addition to the physical properties shown in Table 1 995-1000mm (the difference of maximum and the minimum value is 5mm) and thickness were almost the same as the configuration of the building envelope of a secondary

mold, the periphery front face excelled [ thickness ] in 98-101mm (the difference of maximum and the minimum value is 3mm), and abbreviation homogeneity at smooth nature, and 0.06g /, length cc, and width had the very beautiful consistency.

[0022] Since primary cracking severity is as small as 50% in the example 1 of a comparison, although there are not a leak of foam and deformation according to the result of Table 1, the diameter of average air bubbles is too (it is not a desired thing.) small, 25% compressive stress is small, and the rate of continuous foam is also low, and 25% compression set is also large.

Although it was satisfied with the examples 2 and 3 of a comparison of the physical properties of the last foam, the leak of foam and deformation produced all. On the other hand, in the examples 1 and 2, there is such no fault and the efficiently excellent engine-performance article has been manufactured.

[0023] In examples 3-4 and four to example of comparison 5 this example, the relation between a secondary molded dimension and the foam dimension immediately after the completion of secondary foaming expansion is examined. That is, it is processed on the same conditions as an example 1 using primary metal mold with the predetermined molded dimension which shows the specified quantity (shown in Table 2.) of the same admixture as an example 1 in Table 2.

Furthermore, the same conditions as an example 1 and a facility performed secondary foaming. In addition, the magnitude of a secondary molded dimension is shown in Table 2. These results are also shown in Table 2.

[0024]

[Table 2]

表 2

		実施例3	実施例4	比較例4	比較例5	
1次型発泡性混和物仕込量 (Kg)		5. 5	6. 7	5. 2	7. 1	
一次型寸法 [A] (mm)	縦	398	425	391	434	
	横	398	425	391	434	
	高さ	39	41	38	42	
二次型寸法 [B] (mm)	縦	1000	1000	1000	1000	
	横	1000	1000	1000	1000	
	高さ	100	100	100	100	
2次発泡終了 直後の発泡体寸法 [C] (mm)	縦	1018	1087	1002	1123	
	横	1025	1090	993	1111	
	高さ	102	109	100	112	
$\frac{\text{二次型縮小率} (\%) }{[C] - [B]}$	縦	1. 8	8. 0	2. 0	11. 0*	
	横	2. 4	8. 3	-7. 0*	10. 0	
	高さ	2. 0	8. 3	0*	10. 7*	
	平均	2. 1	8. 2	-1. 7*	10. 6*	
最終発泡体寸法 バラツキ (mm)	縦	最大値	970	1038	958	
		最小値	964	1035	949	
		差	6	3	9	
	横	最大値	975	1039	959	
		最小値	972	1037	940	
		差	3	2	19	
	高さ	最大値	98	106	95	
		最小値	95	103	88	
		差	3	3	7	
最終発泡体の取り出しの容易性		容易	容易	容易	困難	
最終発泡体の割れ発生の有無		無し	無し	無し	有り	

[0025] In Table 2, the foam dimension immediately after the completion of secondary foaming expansion [C] is compulsorily taken out from metal mold, without cooling the foam immediately after the completion of secondary foaming, measures the dimension of three places each about each of length, width, and height, and expresses it with the average. Moreover, the variation in the last foam dimension measures the maximum part and the minimum part about the length of the foam after cool down ready, width, and height within a secondary mold, respectively, and expresses them with the difference. It separates from the numeric value which attached \* mark

in Table 2 from the range of the 3rd invention.

[0026] According to the above result, as shown in Table 2, in the examples 4 and 5 of a comparison with unsuitable secondary mold reduction percentage, the dimension variation of the last foam was large. Moreover, in the example 5 of a comparison, the crack with a die length [ of 200mm ] and a depth of 10mm occurred on the foam top face, and drawing from the mold of the last foam is stiff, and needed to take out fairly by force. On the other hand, in the examples 1, 3, and 4, there were no above faults (it is very few although there is variation.), and they were good. In addition, in this invention, it is not restricted to what is shown in the above-mentioned concrete example, but can consider as the example variously changed within the limits of this invention according to the purpose and the application.

---

[Translation done.]